Sam Schnider x 2845

NBS IR 82-26/3(R)

DEC 4 - 1982

EA-6010-33 Dist. Category UC-90C

MATERIALS RESEARCH FOR THE CLEAN UTILIZATION OF COAL

Quarterly Progress Report

July - September 1982

S. J. Schneider Project Manager

Center for Materials Science National Bureau of Standards U. S. Department of Commerce Washington, D. C. 20234

PREPARED FOR THE UNITED STATES DEPARTMENT OF ENERGY

Office of Advanced Research and Technology

Under Contract No. EA-77-A-01-6010

"This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights."



### TABLE OF CONTENTS

	MENTAL OF PRODUCED TO DITTE	PAGE
ı.	SUMMARY OF PROGRESS TO DATE	1
II.	DETAILED DESCRIPTION OF TECHNICAL PROGRESS	2
	1. Materials Performance and Properties	2
	2. Creep and Related Properties of Refractories	3

refraction in the power to releasely the national and become been under

sillowized silliam chialds were supe at traperatures so to 1900 fc

preliminary to corrying out static list testi-

Presenting of Auroscopies (N. J., Tiple, S. L. Hoteniel

I. SUMMARY OF PROGRESS TO DATE

# **Brief Summary**

 Materials Performance and Properties (H. M. Ondik, B. W. Christ, and A. Perloff)

The book, "Construction Materials for Coal Conversion--Performance and Properties Data," has been completed, was published in September and is now available for sale from the Superintendent of Documents, Government Printing Office.

 Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, and S. M. Wiederhorn)

Unit creep measurements were made on the five fused cast oxide refractories in order to identify the maximum use temperatures under static stress conditions. The materials were tested up to 1400 °C using stresses of 10 to 30 MPa (1.4 to 4.4 kpsi). Strength measurements of siliconized silicon carbide were made at temperatures up to 1400 °C preliminary to carrying out static load tests.

# Brief Supply

Margardal's Performance out Properties (N. M. Oneth., D. M. Covict.,

The pool, "Construction Materials for Coal Conversion-Performance and Franceties Data," has been completed, was sublished in September and is wow available for sale from the Superintendent of Converses, Constructs Painting Office.

Group and Delates Properties of Resentations (N. J. 7198e, C. L. McDensel,

Unit presponessurements were made on the five fines cast mide refrectories in order to identify the maximum use comparation and editions that materials were tested up to 1400 °C using stressed of 10-to 30 PPs-(1.4 to 4.4 tpsi). Strength resourances of siltennias siltenn carbide were made at temperatures up to 1400 °C using preliminary to carrying out static test trate.

- II. DETAILED DESCRIPTION OF TECHNICAL PROGRESS
- Materials Performance and Properties (H. M. Ondik, B. W. Christ, and A. Perloff)

### Progress:

The book, "Construction Materials for Coal Conversion--Performance and Properties Data," has been completed. During the last quarter, the various incomplete subsections were finished and reviewed. The total manuscript was revised, reviewed by the publications division and submitted for publication. The printed books, with issue date September, 1982, are in hand and available for purchase. The book is a National Bureau of Standards Special Publication, number 642. NBS SP642 is available under stock number 003-003-02442-2 from the Superintendent of Documents, Government Printing Office, Washington, DC 20402, for \$16.00 (add 25% for mailing outside the U.S.).

1200, and 1600 %: The results are shown in Table 3. The

II. USTAILED DESCRIPTION OF TECHNICAL PROCRESS

Materials Performance and Properties (N. M. Ondie, B. M. Christ, and A. Perlorf)

## Progress:

The book, "Construction Meterials for CosT Conversion-Performance and Properties Data," has been completed. During the Jast querter, the various incomplete subsections were finished and reviewed. The total manuscript was revised, reviewed by the publications division and subsitted for publication. The printed books, with issue data September, 1862, are in hand and available for publication, number 642. NES SPEAZ is available of Standards Special Publication, number 642. NES SPEAZ is available under stock number, 003-003-02482-2 from the Superintendent of Documents, for mailing outside the U.S.).

 Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, and S. M. Wiederhorn)

### Progress:

Failure stresses and temperatures for the five fused cast oxide refractories were determined under conditions of unit creep. In this technique specimens were held under constant stress for periods of 24 hours at temperatures starting at 800 °C; and, then the temperature was raised 100 °C each 24 hours until excessive creep or failure by crack propagation occurred. Measurements were made using stresses of 10, 20 and 30 MPa for each of the materials. The results of the tests are summarized in Tables 1 and 2. The most creep resistant materials were the  $\alpha$  alumina and the chrome alumina spinel, as determined from the deflection measurements.

The microstructures of two fused cast materials are shown in Figure 1. The varying grain size and porosity in the materials are clearly seen in these light micrographs. Thin sections of the materials were made for examination by electron microscopy in order to identify the phase distribution within the compacts.

Some preliminary tests were carried out on a siliconized silicon carbide material. The billet was one of five received from the Norton Company and designated NC-435. The billet was 10 x 10 x 0.6 cm and was cut into bars 4 x 5 x 50 mm for testing in four-point bending mode.

Sets of 3 bars were broken at room temperature and after holding for one hour at 1200, 1300, and 1400 °C. The results are shown in Table 3. The strength decrease at 1400 °C occurred, of course, because the silicon space filling component melted. Spheres of melted silicon were found on

. Creep and Related Properties of Refrectories (s. J. Tighe, C. L. McDoniel, and S. M. Miederhorm)

## Zusypowy.

Failure stresses and temperatures for the first fund cast calds rearractories were determined under conditions of unit crees. In this technique specimens were held under constant stress for reviods of 24 hours at temperatures starting at 800 °C; and, then one temperature was release 100 °C each 24 hours until excessive crees or failure by crack propagation occurred. Measurements were made using virtuals of 10, 20 and 30 My; for each of the most creen made using virtuals of 10, 20 and at the fairs are suggested in Tables 1 and 2. The most creen resistant extentials when deflection measurements.

The microstructures of two fuses cast mountains are shown in Figure 1. The varying grain size and porosity in the materials ore clearly seen in these light micrographs. This sections of the materials when made for examination by electron microscopy in order to identify the phase distribution within the concects.

Some preliminary tests were carried out on a sittennial silten carbido material, The billet was one of five meeted from the Worten Company, and designated MC-435. The billet was 10 x if x 0.5 cm and was out into hers 4 x 5 x 50 cm for testing in four-point bending mode.

Sets of 3 bers were broken at room temperature and atter moiding for one moor at 1200, 1300, and 7000 °C. The results are those in Table 3. The strength decrease at 1400 °C occurred, of course, business in Table 3. The space filling component melted. Sphere at motors to over found on

the fracture surface of specimens broken at 1400 °C. The silica film that formed on the exposed ground surfaces during the soaking period prevented the phase from extruding through these surfaces.

Specimens were held under a static load for 168 hours to determine any creep or strength loss as a result of stress. As seen from the tabulation in Table 4 there is a slight strength increase. There was no measurable creep during this period.

### Plans:

A final report on the fused cast and silicon carbide materials will be prepared and will include the creep results as well as the microstructural analyses.

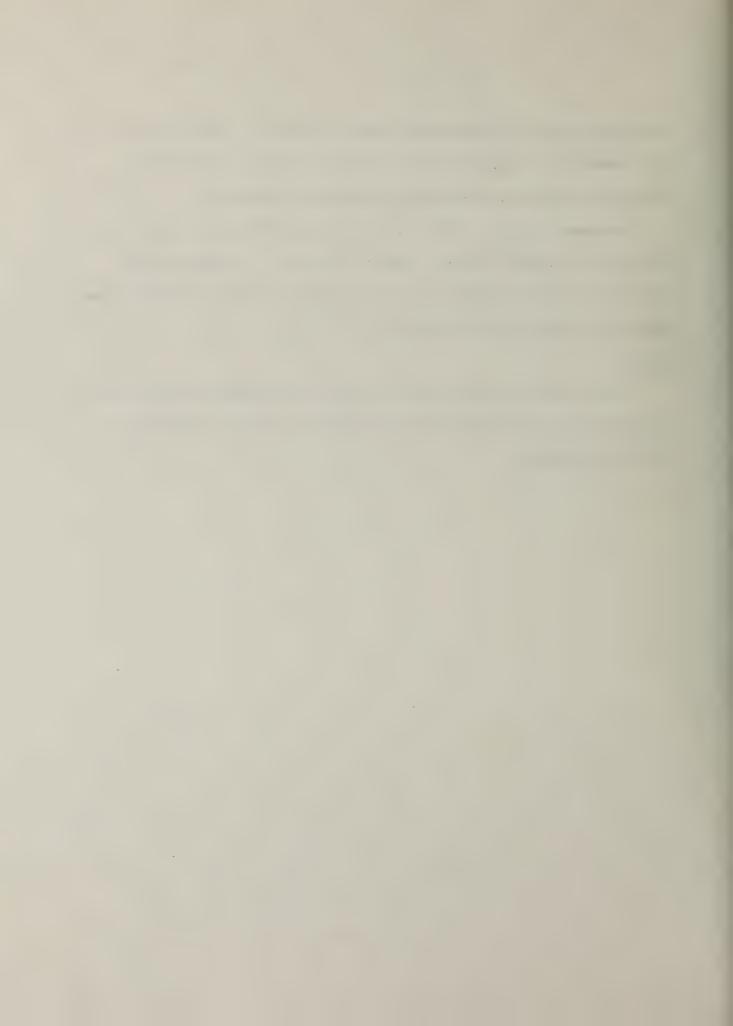


Table 1. Summary of strength obtained with dynamic load.

Material	Modulus of Ru MPa	
	25 °C	1000 °C
Al <sub>2</sub> O <sub>3</sub> ·ZrO <sub>2</sub> ·SiO <sub>2</sub> Monofrax CS-3	93.0 <u>+</u> 7.5	109.0 <u>+</u> 13.8
•	25 °C	1100 °C
α + β Al <sub>2</sub> O <sub>3</sub> Monofrax M	38.1 <u>+</u> 1.9	36.2 <u>+</u> 3.0
α Al <sub>2</sub> O <sub>3</sub> Monofrax A	64.6 ± 5.6	52.3 <u>+</u> 1.8
Cr-Al Spinel Monofrax E	37.0 <u>+</u> 2.4	47.5 <u>+</u> 4.3
Cr-Al Oxide Monofrax K-3	42.2 <u>+</u> 3.4	27.3 <u>+</u> 10.0

Table 2. Failure temperature for samples under static load.

Material	Static Load 10 MPa Failure Temperature °C	Static Load 20 MPa Failure Temperature °C	Static Load 30 MPa Failure Temperature °C
Al <sub>2</sub> O <sub>3</sub> •ZrO <sub>2</sub> •SiO <sub>2</sub> Monofrax CS-3	900	900	800
α + β Al <sub>2</sub> O <sub>3</sub> Monofrax M	1400	. 1100	*
α Al <sub>2</sub> O <sub>3</sub> Monofrax A	1300	1300	*
Cr-Al Spinel Monofrax E	1400	1100	*
Cr-Al Oxide Monofrax K-3	1400	: 1100	*

<sup>\*</sup>In progress.

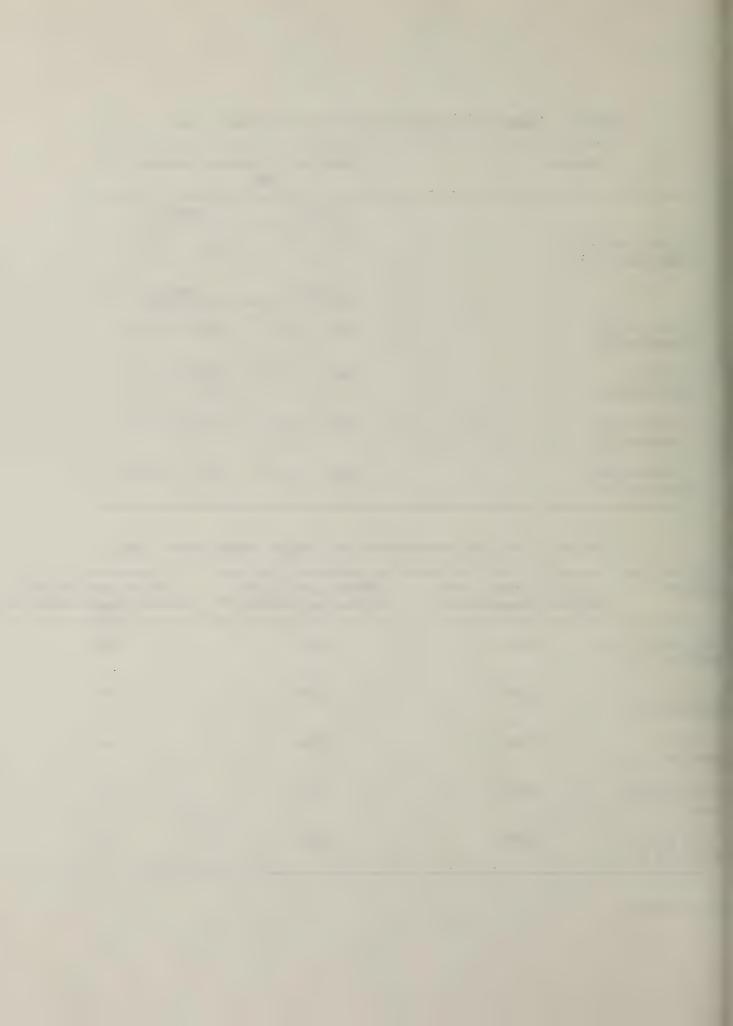
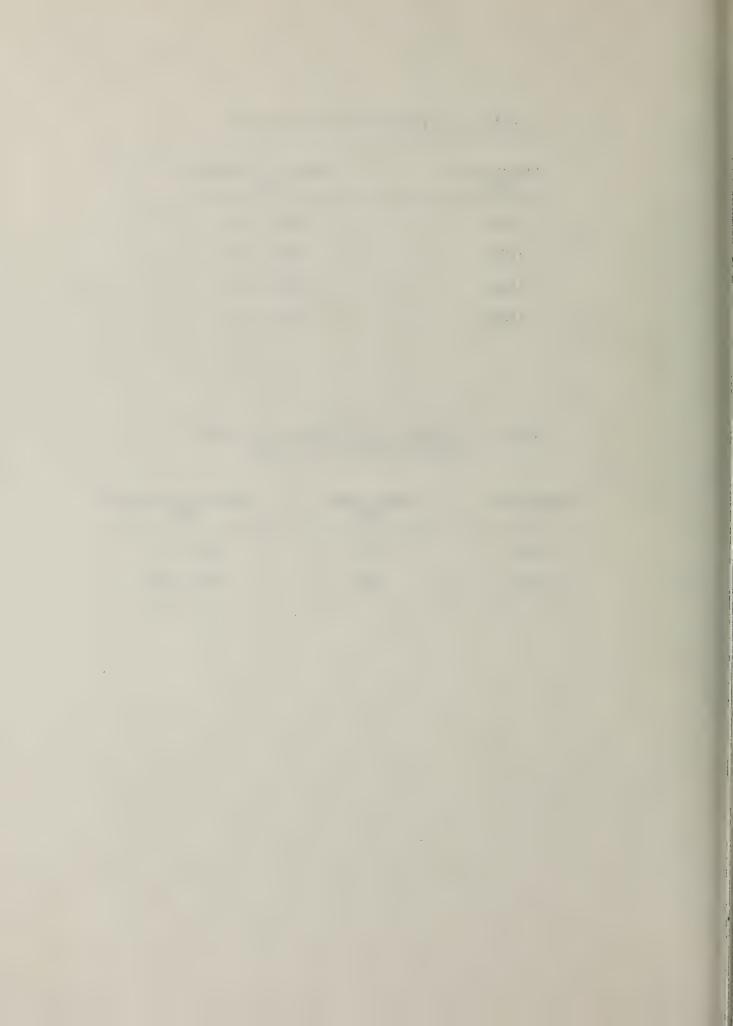


Table 3. Strength of Siliconized SiC

Temperature °C	Modulus of Rupture MPa
25	367 <u>+</u> 74
1200	439 <u>+</u> 47
1300	<b>376</b> ± 56
1400	91 <u>+</u> 30

Table 4. Strength of siliconized SiC after 168 hours under static load.

Temperature °C	Static Load MPa	Modulus of Rupture MPa
1200	100	481 <u>+</u> 59
1300	100	394 <u>+</u> 38





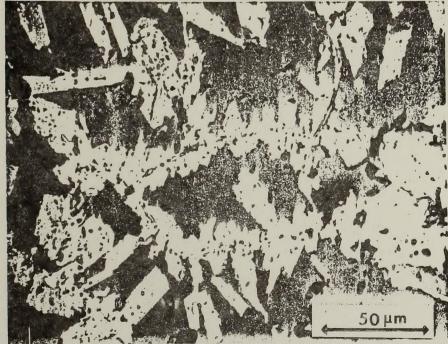


Figure 1. Light micrographs of polished sections of fused cast refractory samples. (a) alumina-zirconia-silica, showing plates of alumina and alumina containing zirconia in an alumina-silica glass; (b) chrome-alumina showing plates of chromia in a chrome alumina matrix.



Figure 1. Light micrographs of political sections of four case entractant samples. (a) simulna-circonia-silica, encerno cista en alumina and alumina containing sincents on an elumina-citica glasse. (b) chrome-lightenia choule planes of chrome in a



